Development of Ultra-High Mechanical Damping Structures Based on the Nano-Scale Properties of Shape Memory Alloys

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14. ABSTRACT

We recently discovered that micro and nano pillars milled by Focused Ion Beam (FIB) on Cu-Al-Ni shape memory alloys (SMA) exhibited excellent superelastic behavior and ultra-high mechanical damping at small scale. This behavior offers an interesting potential for mechanical damping applications in Micro Electro-Mechanical Systems and other applications. The objective of the present project was to further explore the ultra-damping behavior of micro/nano pillars and arrays of such features. Several alloys of Cu-Al-Ni SMA, with the appropriate composition to exhibit superelastic effect at room temperature, were induction melted, and then oriented single crystals were grown from these alloys. Many different sizes of micro-nano pillars were milled on slides cut from the oriented single crystals, by FIB. The superelastic effect of these pillars was subjected to nano-compression tests using an instrumented nanoindenter, and the damping coefficient was measured from the load-displacement curves. A high damping with a loss coefficient of about $\eta=0.16$ has been confirmed to be stable over 100s of cycles. In order to verify the reliability of the Cu-Al-Ni pillars for mechanical damping applications, arrays of micro pillars were milled in a similar way. Nanocompression superelastic behavior was tested along with cyclic behavior, obtaining a perfectly recoverable behavior above more than 2000 cycles, with a loss coefficient $\eta>0.1$. Study of the pillars from arrays showed good reproducibility. Finally, a study of the scalability of the production process was realized and the required methodology to produce very large arrays of pillars by using the VLSI techniques employed in microelectronics was analyzed. It can be concluded that micro-nano pillars of Cu-Al-Ni SMA offer a good potential for mechanical damping applications at small scale, but the development of very large arrays and structures or stacks of arrays of pillars has to be still optimized.

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Summary

In recent years it was discovered that micro and nano pillars milled by Focused Ion Beam (FIB) on Cu-Al-Ni Shape Memory Alloys (SMA) exhibited a good completely recoverable superelastic behavior and an ultra-high mechanical damping at small scale. This behavior seems to offer an interesting potential for mechanical damping applications in Micro Electro-Mechanical Systems and eventually in many applications like surface shaking damping. Then the objective of the present project was to explore the real capabilities of damping applications of micro-nano pillars and arrays of such features for creating structures with ultra-high damping.

To develop the project, several alloys of Cu-Al-Ni SMA, with the appropriate composition to exhibit superelastic effect at room temperature, have been induction melted, and then [001] oriented single crystals were growth from these alloys. Many different size of micro-nano pillars have been milled on slides cut from the oriented single crystals, by FIB.

The superelastic effect of these pillars has been tested by Nano-compression tests by using an instrumented nanoindenter Hysitron TI-950, and the damping coefficient has been measured fro the load-displacement curves. A high damping with a loss coefficient of about $\eta=0.16$ has been confirmed to be stable over hundred of cycles.

In order to verify the reliability of the Cu-Al-Ni pillars for mechanical damping applications, arrays of micro pillars have been milled in a similar way. Nano-compression superelastic behavior has been tested along cycling, obtaining a perfectly recoverable behavior above more than two thousand cycles, with a loss coefficient $\eta>0.1$. In addition a systematic study of the all the pillars from the arrays shows a good statistics in what concerns the reproducibility from the different pillars of an array.

Finally a study of the scalability of the pillars production process has been realized and the required methodology to produce very large arrays of pillars by using the VLSI techniques employed in microelectronics has been analyzed.

It can be concluded that micro-nano pillars of Cu-Al-Ni SMA offer a good potential for mechanical damping applications at small scale, but the development of very large arrays and structures or stacks of arrays of pillars has to be still optimized.

A. Introduction

The present project arose from previous works in collaboration between the University of the Basque Country (UPV-EHU) and the Massachusetts Institute of technology (MIT). During these works a good shape memory and superelastic behavior was observed micro and nano pillars milled by focused ion beam in single crystals of Cu-Al-Ni shape memory alloys [1], in which an ultra-high damping was observed as a consequence of a size effect at nano-scale [2]. Then the MIT applied for a USA patent [3] concerning this ultra-high damping behavior. Some further analysis showed that Cu-Al-Ni SMA could exhibit a better behavior than the commercial Ti-Ni SMA at nano-scale [4, 5].

Then, the general objective of the present EOARD project was to explore the potential capabilities of such SMA materials to develop Ultra-High Mechanical Damping Structures by using their nano-scale properties.

B. Experimental Approach

The methodology originally proposed cover several key aspects, which are summarized as follows:
1.- Production of the SMA materials.
2.- Milling of micro pillars and arrays of micro pillars.
3.- Testing the damping behavior at nano-scale of such micro-structures.
4.- Analyze the scalability of such microstructures for very large scale production.
5.- Design the methodology for measuring the dissipated energy in large structures.

C. Accomplished results

1.- Production of the SMA materials.
We have produced the Cu-Al-Ni SMA with the appropriate composition to undergo the martensitic transformation below RT, and consequently exhibiting superelastic effect at RT.
The alloys were produced from high purity raw metals, by induction melting under vacuum and centrifugal casting.
Then oriented [001] single crystals were growth by Bridgman method with oriented seed in graphite crucible. These crystals were thermally treated for 1800 s at 900ºC and quenched in iced water in order to freeze the high temperature beta phase, to allow further stress-induced martensitic transformation. From these single crystals, thin slides were cut and mechanically polished, as the starting material to produce the micro/nano structures.

2.- Milling of micro pillars and arrays of micro pillars.

The oriented single crystal slides have been used to produce micro-nano structures of pillars. We have produced micro and nano pillars in a large range of size from 1.5 μm to 400 nm, by focused ion beam in a FEI Dual Beam Helios 650, in order to test their damping behavior. See Figure 1 as an example of such micro-nano pillars.
In a second step, we have optimized the methodology for producing arrays of micro-pillars by FIB, in order to check the reproducibility of the mechanical damping behavior

in a series of pillars, and also to test the long cycling behavior. Different arrays of pillars have been produced, with different number and shape of pillars. In Figure 2 an example of square micro-pillars array is shown.
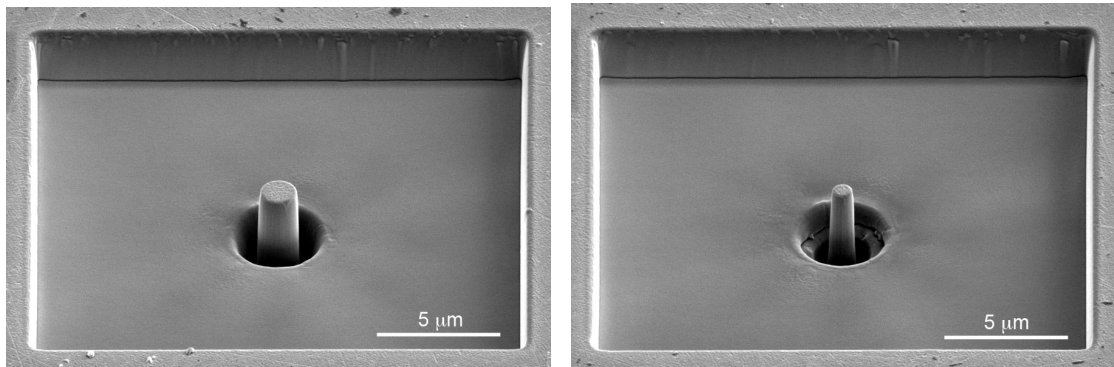

Figure 1. Two examples of cylindrical micro pillars milled by FIB with different diameter, to test the size influence on the nano-compression behavior.
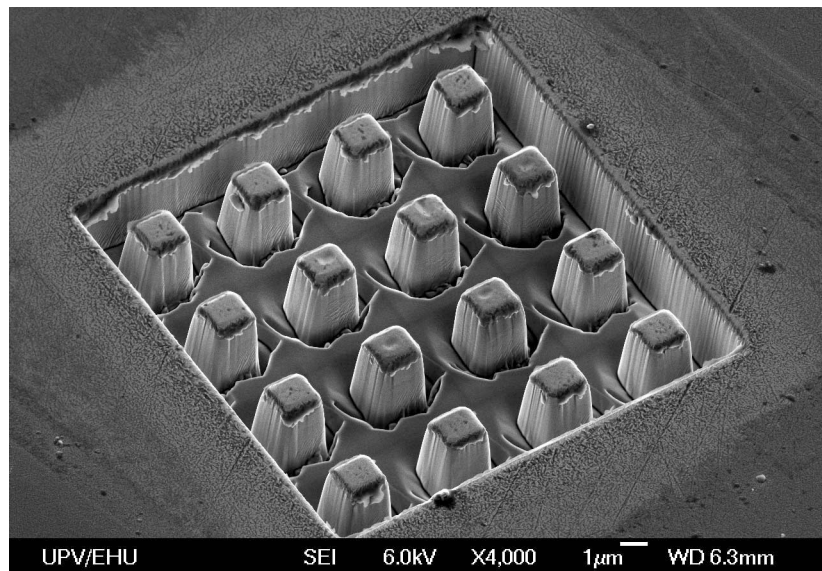

Figure 2. Array of square pillars milled by FIB on a [001] single crystal slide of Cu-Al-Ni shape memory alloy, to systematically test, by nano-compression, the reproducibility of the superelastic behavior, in order to verify the reliability of the mechanical behavior. In the present image the indent produced by the sphero-conical indenter during the nano-compression tests can be seen on the top of some pillars, whiles the others are still flat before testing.

3.- Testing the damping behavior at nano-scale.

One important point from the beginning of the project was to verify whether the ultra-high damping exhibited by the pillars is stable on cycling or simply disappears after some superelastic cycles. So, we approached the study of the superelastic nano-compression behavior of the micro pillars along cycling at different loading rates, this means different frequencies.

We observed a training effect along cycling in which the critical stress for stress-induced transformation decreases as a consequence of the development of nucleation points for martensite at atomic scale, Figure 3 and 4. However, in spite of the clear evolution of the superelastic behavior on cycling over hundreds of cycles, locally the cycles become quite reproducible for the same maximum load and loading rate.
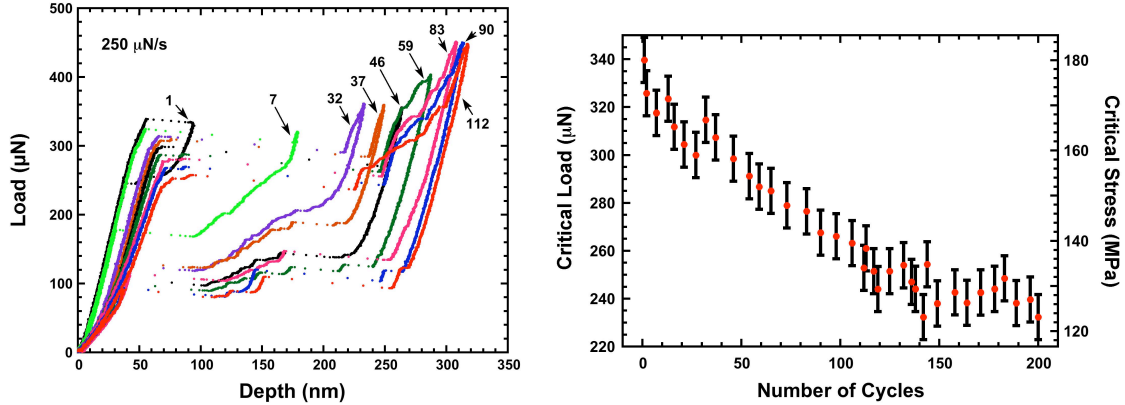

Figure 3 & 4: Evolution of superelastic nano-compression cycles along cycling and the corresponding evolution of the critical stress for stress-induced transformation [6].

The influence of the loading rate (cycling frequency) was also studied in an order of magnitude, from 100 μNs^{-1} to 1000 μNs^{-1}, and the corresponding signatures analyzed and discussed. One of the most important points is that the mechanical damping behavior becomes stable after the first 50 training cycles, Figure 5, reaching an stable value of the loss factor about $\eta=0.16$, which is really a very high mechanical damping.
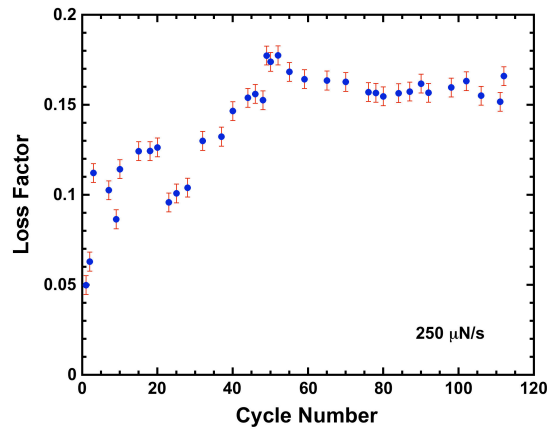

Figure 5. Evolution of the damping coefficient along cycling in a micro pillar [6]. There is an increase during the first 50 cycles and then the loss factor become stable.

This is the first time that the superelastic cycling behavior in SMA was approached at nano scale, and this study was published in Acta Materialia [6], a copy of this publication is attached.

However, with view on mechanical damping applications, is not enough that the material undergoes some hundred of cycles and so in a second step we study the reliability of the pillars along thousand of cycles. Then we performed a series of tests on each individual pillar from the arrays shown in Figure 2. In Figure 6 the reproducibility of five superelastic cycles after one thousand previous cycles is clearly seen, evidencing a good behavior along cycling.
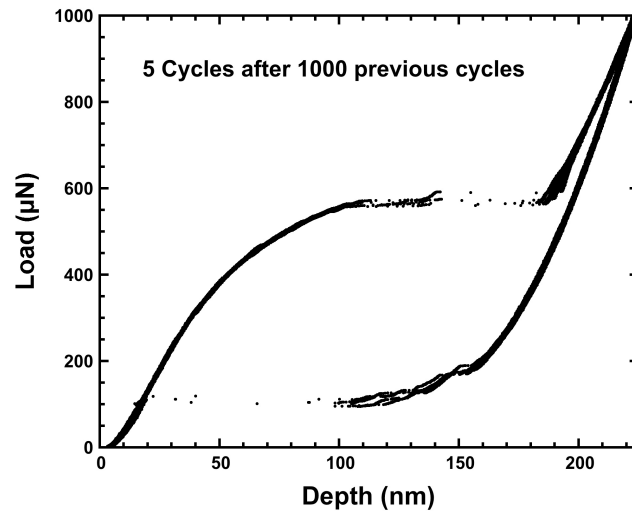
Figure 6. Five superelastic consecutive cycles measured on a pillar from the array of figure 2, after one thousand previous cycles. The reproducibility is remarkably good.

This kind of study has been systematically performed on all the pillars of the array from Figure 7, in order to obtain a statistic of the superelastic behavior and the mechanical damping for the pillars. In addition some pillars were tested up to two thousand cycles, as show in Figure 8, evidencing an exceptional good reproducible and reliable behavior as well as a high mechanical damping.
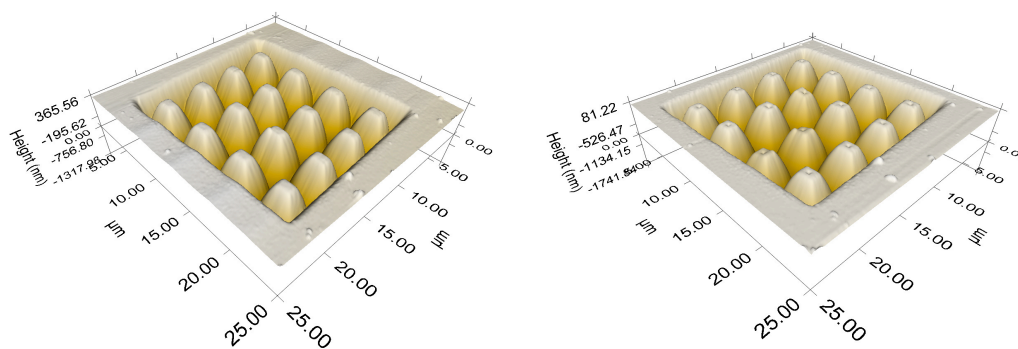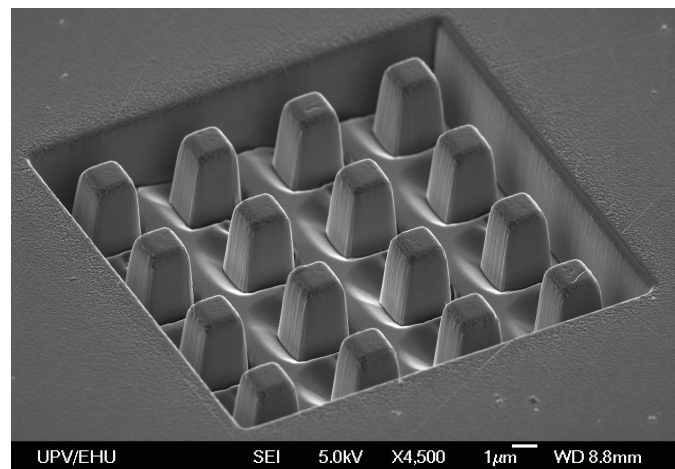

Figure 7. SEM image of another array of square pillars used for systematic testing of all pillars by nano-compression. The images below correspond to the scanning images taken with the indenter before and after testing the pillars.
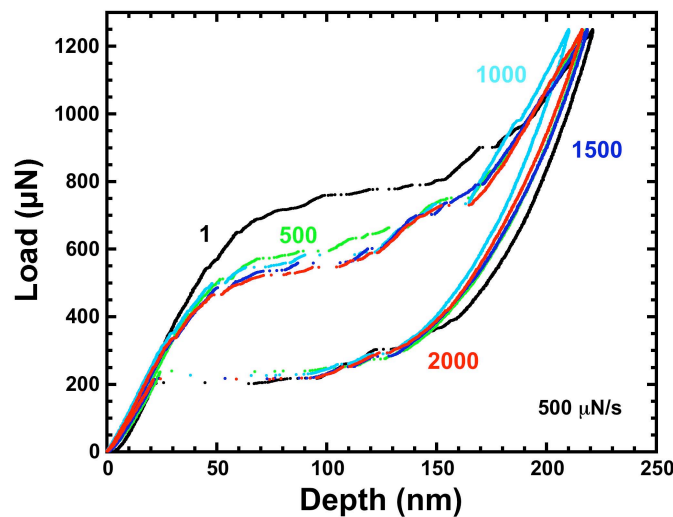
Figure 8: Superelastic nano-compression cycles on a pillar from the array of figure 7. There is an initial training, as previously observed, and then the superelastic behavior, as well as de associated damping, become reproducible during more than 2000 cycles, till the maximum number of cycles applied with the nano-indenter. The loss factor is $\eta > 0.1$ along cycling.

These studies are not published yet, but Prof. J. San Juan has been invited to give a plenary lecture about this topic in the Gordon Research Conference on "Nano-mechanical Interfaces" in Hong Kong [7]. Traditionally in this series of Conferences, unpublished results are presented, and consequently these results will be published just after the meeting.

4.- Analyze the scalability of such microstructures for very large scale production.

It was expected that in the frame of the project, the production of large arrays of pillars would be produced by lithographic techniques as used for microelectronics or MEMS production. However, although most of the required steps are possible, there are some inherent problems that we have not been able to overcome. In the following we describe the complete analyzed process analyzing the difficulty of specific steps.

a) Integrating the single crystal slides on silicon wafers.
This is feasible by diffusion bonding. This could be performed by evaporating a thin film of gold and thin on both sides of the joining interface, the wafer and the single crystal of Cu-Al-Ni, and then applying pressure and temperature (>230ºC) under vacuum. We started the fabrication of the diffusion bonding equipment with the required performances, but due to the financial restrictions in Spain it is not yet operative.

b) Micro-Lithography of the large arrays of micro-nano pillars.
This step does not offer particular difficulties and several techniques could be used: classical optical lithography, interferential lithography or e-beam lithography. We are ready to apply the last two techniques that are the more appropriate for small features.

c) Etching of the micro-nano pillars

This is the main drawback of the process, which we have not been able to overcome yet. To produce pillars or other features with a aspect ratio above three and good lateral sides, a Directional Reactive Ion Etching (DRIE) has been considered. However, the SMA used in the present work are cooper based, Cu-14Al-4Ni (wt%) so they contain at about 82 wt% Cu and from the point of view of etching, they will behave close to pure Cooper.

Etching of cooper is rather difficult because the most used chlorine etching produce non-volatile cooper components, which remain on the surface. To avoid that more complex formulations are used between 180ºC and 230ºC, and from the literature, we considered as optimal process the use of   at   , because the use of higher temperatures during the long required time, could destroy the alloy itself by diffusion processes. This high temperature etching require the use of specific high temperature resistant resist during the lithographic process, and this resist became very difficult to remove after a high temperature curing as the required for the cooper-based alloy etching. Indeed, after such curing the resist cannot be chemically removed and we have to use another plasma etching.

At present we have not found the right combination of process and the laboratory to do the systematic tests to optimize the process. It was expected to do it at the Micro Technologies Laboratory (MTL) of the MIT, but the etching equipment of this laboratory was dedicated equipment for specific processes, which not allow the use of our alloys that risk contaminating the system. We are still looking to solve this etching procedure.

As an alternative solution we have also considered the production of large pillars array by Focused Ion Beam, but this cannot be performed in a Dual Beam system like the one used to produce the small arrays. Indeed, the milling of uniform features over large areas require specific FIB milling systems like, for instance, the Ion-Line produced by the Raith company from Germany. We are moving in this direction and talked with the responsible of this company to perform some tests, but we have still to establish a cooperation agreement.

5.- Design the methodology for measuring the dissipated energy in large structures.

The methodology for testing intermediate arrays is already ready. A new flat punch indenter for the high load head of the Hysitron TI-950 Nano-Indenter has been bought to perform compression tests on 100x100 pillar arrays (10.000 pillars), just approaching the limit of the 10 Newton loading capabilities of this high load head. Beyond this size of the array of pillars, a high precision testing machine will be required, and probably the best adapted would be the Electro-pulse series from INSTRON or the Electro-force series from BOSE Corporation. Obviously such equipment offers enough load, but they have not the displacement resolution required to measure the superelastic cycle of a layer of pillars, so a scalability to bigger pillars would be required (accepted).

However this equipment would be able to measure the mechanical damping due to superelastic behavior of multilayer stacks of single crystal slides produced by diffusion bonding.

D. Conclusions

From the above presentation and the whole set of results obtained along the project, we may conclude that:

- Micro and nano pillars of Cu-Al-Ni shape memory alloys exhibit a very high damping behavior over thousand of cycles, without apparent degradation.
- The damping loss factor measured for many different pillars is in between $0.10 < \eta < 0.16$. This means that approximately the dissipated energy is between 30% and 50% of the maximum applied energy per cycle and unit volume.
- Arrays of pillars show a very reproducible behavior of different pillars, so the concept of damping by nano-compression of a pillar can be extrapolated to large arrays of pillars.
- The methodology to develop large arrays of pillars by the VLSI techniques used in micro electronics, seems to be feasible, but it is not yet mastered.
- The methodology for evaluating the damping behavior of large arrays of pillars or stacks of arrays has been already designed.

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